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P036238GB:HRG

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08479958001

If the applicant is a corporate body, give the
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DIAMOND

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Carpmaels & Ransford

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Carmichael & Ransford

12. Name and daytime telephone number of person to contact in the United Kingdom

Hugh Goodfellow

020-7242 8692

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BACKGROUND OF THE INVENTION

This invention relates to a method of marking or fingerprinting a diamond material, in particular a single crystal synthetic diamond material produced by chemical vapour deposition (hereinafter referred to as CVD), thereby providing a mark of origin or fingerprint in the diamond material, or a means by which its synthetic nature can more easily be determined.

Methods of depositing material such as diamond on a substrate by CVD are now well established and have been described extensively in the patent and other literature. Where diamond is being deposited on a substrate, the method generally involves providing a gas mixture which, on dissociation, can provide hydrogen or a halogen (e.g. F,Cl) in atomic form and C or carbon-containing radicals and other reactive species, e.g. CH_x , CF_x wherein x can be 1 to 4. In addition, oxygen containing sources may be present, as may sources for nitrogen, and for boron. Nitrogen can be introduced in the synthesis plasma in many forms; typically these are N_2 , NH_3 , air and N_2H_4 . In many processes inert gases such as helium, neon or argon are also present. Thus, a typical source gas mixture will contain hydrocarbons C_xH_y wherein x and y can each be 1 to 10 or halocarbons $\text{C}_x\text{H}_y\text{Hal}_z$ wherein x and z can each be 1 to 10 and y can be 0 to 10 and optionally one or more of the following: CO_x , wherein x can be 0,5 to 2, O_2 , H_2 , N_2 , NH_3 , B_2H_6 and an inert gas. Each gas may be present in its natural isotopic ratio, or the relative isotopic ratios may be artificially controlled; for example hydrogen may be present as deuterium or tritium, and carbon may be present as ^{12}C or ^{13}C . Dissociation of the source gas mixture is brought about by an energy source such as microwaves, RF (radio frequency) energy, a flame, a hot filament or jet based technique and the reactive gas

species so produced are allowed to deposit onto a substrate and form diamond.

CVD diamond may be produced on a variety of substrates. Depending on the nature of the substrate and details of the process chemistry, polycrystalline or single crystal CVD diamond may be produced.

The development in the level of sophistication of methods of producing CVD single crystal diamond has meant that this material is becoming increasingly more suitable for use in industrial applications or in ornamental applications such as synthetic gemstones. However, in many applications there is a need to provide a method of determining the source of synthetic diamond used in these applications in order to verify its origins or synthetic nature.

SUMMARY OF THE INVENTION

According to one aspect of the invention, there is provided a method of incorporating a mark of origin, such as a brand mark, or fingerprint in a CVD single crystal diamond material, which includes the steps of providing a diamond substrate, providing a source gas, dissociating the source gas thereby allowing homoepitaxial diamond growth, and introducing in a controlled manner a dopant into the source gas in order to produce the mark of origin or fingerprint in the synthetic diamond material, which dopant is selected such that the mark of origin or fingerprint is not readily visible or does not affect the perceived quality of the diamond material under normal viewing conditions, but which mark of origin or fingerprint is visible or rendered visible under specialised viewing conditions, such as when exposed to light or radiation of a specified wavelength, for example.

The mark of origin or fingerprint is preferably provided in the form of one or more layers or regions grown into the diamond material during synthesis.

Such a mark of origin or fingerprint in a CVD single crystal diamond material is most appropriate in CVD diamond which is of high commercial or gem quality. Synthesis of such high quality CVD diamond is best performed using a diamond substrate having a surface on which growth takes place which is substantially free of crystal defects, and this forms a preferred version of the method of the invention.

In one embodiment of the method of the invention, the dopant is nitrogen, which produces a mark of origin or fingerprint, preferably in the form of a layer, that shows 575 nm and/or 637 nm luminescence peaks, with their associated vibronic systems, under suitable shorter wavelength excitation. The nitrogen doped layer may also show a photoluminescence line at 533 nm.

In an alternative embodiment of the invention, the dopant is a combination of nitrogen and boron, where the boron is preferably present in a higher concentration than the nitrogen, which produces a mark of origin or fingerprint, preferably in the form of a layer, that generates characteristic phosphorescence, peaking at about 400 nm, under suitable shorter wavelength excitation.

In a particularly preferred embodiment of the invention, a combination of layers that generate 575 / 637 nm luminescence and 400 nm phosphorescence under suitable shorter wavelength excitation is grown into the diamond material during synthesis.

In accordance with a further aspect of the invention there is provided a CVD single crystal diamond material bearing a mark of origin or fingerprint in the bulk thereof, which mark of origin or fingerprint is grown into the diamond material during the synthesis thereof, preferably in accordance with the method described above.

The single crystal diamond material may be prepared for a range of industrial applications, in particular those in which the synthetic diamond is

a visible element to the user, or where the synthetic diamond element is re-useable or needs periodic reprocessing, as is for example the case with diamond cutting blades such as scalpels.

Alternatively, the single crystal diamond material may be prepared or suitable for preparation as a synthetic gemstone.

The invention also extends to a viewing apparatus for viewing the mark of origin or fingerprint in a CVD single crystal diamond material, object or synthetic gemstone, the apparatus comprising:

a source of light or radiation of a particular wavelength for causing excitation of the mark of origin or fingerprint, resulting in luminescence and/or phosphorescence thereof; and

a viewer for viewing the luminescence and/or phosphorescence.

The apparatus preferably comprises a range of optical filters for viewing the wavelengths emitted by the mark of origin or fingerprint, and means for excluding background white light. The characteristic luminescence and/or phosphorescence may be viewed in the form of a special image detectable directly in the diamond material, or it may be viewed by using instrumentation such as a charge coupled device (ccd) or imaging device such as a digital camera. Alternatively, the luminescence and/or phosphorescence may be characterised by a spectroscopic device such as one or more specific band pass filters and/or frequency specific sensors, or a compact spectrometer. These techniques can be combined, for example using suitable filters in combination with a ccd camera to form frequency specific images.

The apparatus may also include magnification means for magnifying the image of the synthetic diamond material.

DESCRIPTION OF PREFERRED EMBODIMENTS

The invention provides a method of marking synthetic diamond material, in particular such material prepared for use in industrial applications such as cutting tools or as a CVD synthetic diamond gemstone. The method of marking enables the determination of origin, said mark of origin or fingerprint comprising one or preferably more layers grown into the diamond during synthesis, which do not substantially affect the perceived optical or gem qualities of the diamond under normal viewing, or significantly affect any other application specific properties, but which can be viewed under special viewing conditions.

By way of example, diamond scalpel blades are often re-useable, periodically returning to the manufacturer for preparation of a new edge. Used in this application, the mark of origin or fingerprint can fill one or more of the following functions, although its purpose may not be limited to these examples:

- a) Enable the specific manufacturer of the synthetic diamond scalpel to be identified, either by the manufacturer or by the marketplace. This can be used by the manufacturer to ensure that only his own blades are accepted for reprocessing, and improve the ability to track such blades within the reprocessing or in the marketplace at large.
- b) Provide a means by which to generate a distinctive mark, such as a trademark, without degrading the material in its final application. Ordinarily visible identification marks on the synthetic diamond scalpel blade may not be acceptable for some applications because of requirements of hygiene, uniform transparency, or simply market expectation or acceptance.
- c) Enhance the identification of the synthetic nature of the diamond material. Synthetic diamond can offer greater reproducibility and

control in many industrial applications, thus offering a better product.

- d) Provide a means by which modification of the synthetic diamond material may be identified, such modification including changes to physical shape and annealing treatments such as those which modify colour.

By way of further example, in the application of CVD diamond in synthetic gemstones, the mark of origin or fingerprint can fill one or more of the following functions, although its purpose may not be limited to these examples:

- a) Enable the specific manufacturer of the CVD synthetic gemstone to be identified, either by the manufacturer or by the marketplace.
- b) Provide a means by which to generate a distinctive mark such as a trademark.
- c) Enhance the identification of the synthetic nature of the diamond material.
- d) Provide a means by which modification of the CVD synthetic diamond material may be identified, such modification including changes to physical shape and annealing treatments such as those which modify colour.

The exact function of the mark of origin or fingerprint typically determines the form of the mark that is preferred.

In its simplest form the mark may merely comprise a substantial portion of the diamond layer, object or synthetic gemstone, or a single broad layer within it, exhibiting an unnatural characteristic which is only observable under specific artificial conditions of observation and does not significantly

affect the colour of any object prepared from the layer under normal illumination. The obviously artificial element to the mark may result from its colour, possibly in combination with the specific artificial conditions applied in order to observe the colour, or the geometry of the boundaries or distribution of the marked layer within the whole layer, primarily observed as the geometry with which it intercepts the boundaries of the layer, object or synthetic gemstone.

In a more complex form, in order to generate a distinctive mark such as a trademark, the mark of origin or fingerprint generally comprises one or more sets of characteristic layers, either distributed periodically throughout the diamond layer, object or synthetic gemstone or, in the case of a single set of characteristic layers, placed in an appropriate location, generally not too near one of the edges of the object such that minimal removal of material will remove it, nor generally such that the mark is concealed and not observable by components essentially permanently attached to the object in normal use. In the case of a synthetic gemstone, a single set of characteristic layers may be located near the middle of the thickness of the cut stone, or if biased away from the middle then preferably biased towards the table of the cut stone to enable access for viewing when the stone is mounted.

One particular type of layer is one in which the prime active impurity is nitrogen in the form of NV^0 and NV^- centers that generate 575 nm and 637 nm luminescence lines respectively, together with their associated vibronic bands, under suitable shorter wavelength excitation. The combination of these emissions appears yellow/orange and is generally referred to as 'orange luminescence'. Such luminescence extinguishes essentially instantaneously when the excitation source is removed. Whilst clearly visible under suitable artificial illumination conditions, under normal viewing conditions and with appropriately chosen defect concentrations and/or total defect numbers as envisaged in this invention, this type of center does not significantly alter the perceived colour of the diamond.

Another particular type of layer is one in which the prime dominating impurities are boron and a suitable donor such as nitrogen. Donor-acceptor pair recombination may occur in such a layer and the layer then exhibits characteristic broad blue phosphorescence peaking in the region of 400 nm under suitable shorter wavelength excitation. Phosphorescence is visible for a period of time after the removal of the excitation source, the time period typically being several seconds although it can be as long as a minute or more. In this type of layer the nitrogen has two important roles:- providing the donor for donor/acceptor pair recombination, and reducing the B absorption which provides the blue colour in the normal optical band. Provided the absorption attributable to B in the normal optical band is maintained at a sufficiently low level, this center is again clearly visible under suitable artificial illumination conditions, but under normal viewing conditions does not significantly alter the perceived colour of the diamond. Other donors may contribute to this type of luminescence, such as intrinsic defects.

A particularly advantageous arrangement would be where these two types of layer are used alternately or in some other pattern within unmarked material. The shorter wavelength excitation can then be selected to excite both the (575 nm/637 nm) orange luminescence and the (400 nm) blue-band phosphorescence, or a suitable combination of wavelengths can be used.

The method of viewing the mark is in part related to the excitation wavelength(s) used. Using sub-band gap illumination (i.e. light with insufficient energy to excite electrons right across the band gap in diamond, and thus not normally absorbed by diamond itself), the radiation would be transmitted through the volume of the stone, being absorbed only by the defects in the doped marker layers, enabling the volume of the doped marker layers to be excited. Since the wavelengths emitted from the layers are also transmitted by diamond (although some absorption may occur in the defects in the layers) the person viewing the stone would be able to

see, for example by viewing through the table of the stone, a substantial area of the layer which is emitting the colour within the diamond volume.

Since the orange luminescence turns off with the excitation source, the perceived colour of the luminescence from the CVD diamond material, or the coloured layers within it, would change from orange, or some orange/blue combination, to blue. This will be termed the orange/blue flash. Sub band-gap illumination would be the preferred excitation wavelength for simple demonstration of the existence of the mark in the piece of synthetic diamond, allowing its detection through the table of a cut stone even when the layers are significantly further down in the material. In some instances where the material is formed in the shape of a synthetic gemstone it may be preferred to place these layers below the girdle of the stone, so that the edges of the layers are generally concealed by the mount. In other instances it may be preferred to place the layers above the girdle, allowing these edges to be viewed on the crown facets. The closer to the centre of the synthetic stone the layers are positioned, the more difficult it is to remove the mark by repolishing the stone without significant weight loss. The orange/blue flash is particularly characteristic of the combination of these particular marking layers. It provides a unique characteristic not seen in natural diamonds or likely to occur accidentally in synthetic stones. Those skilled in the art will understand that other colour combinations may be possible using other types of defects in the diamond material, and that the invention is not limited to any particular colours and or viewing conditions, but extends generally to any viewable distinctive mark which does not substantially degrade the visual characteristics of the layer or gemstone under normal viewing conditions.

Using above bandgap illumination (i.e. light with sufficient energy to excite electrons right across the band gap in diamond, which thus causes it to be heavily absorbed by diamond itself), the diamond material would itself directly absorb the incoming radiation and limit the penetration depth to near the surface of the stone. This would potentially limit the visibility of the layers to those areas close to the surfaces of the stone which are directly

exposed to the artificial illumination. Two effects may broaden the region in which the illuminating irradiation is absorbed, the first is where the excitation radiation is near bandgap, so that the attenuation of the radiation in the diamond material is rising but is not yet limiting the penetration to a few microns depth at the surface, and the second is where charge carriers excited at the surface by the incoming radiation are able to drift further into the diamond item and then cause excitation of the layers further into the bulk. The ability for this to happen depends on a variety of factors including the general purity and crystal perfection of the synthetic diamond material outside the specifically marked layers.

However, the benefit of using above bandgap radiation is generally to limit the region excited to the surface of the diamond item and thus provide greater definition of the pattern of the layers, although viewing this detail often requires more sophisticated viewing equipment, particularly in view of the thickness of the layers. In this domain, viewing only the exposed edges of the layers and considering the specific example of using alternate orange luminescing and blue phosphorescing layers, both layers would be visible during excitation but only the layers showing blue-band phosphorescence would be visible after the excitation ceased. Above bandgap illumination is particularly useful in a) enabling the detailed geometry of a mark intended as a trademark to be observed, and b) where the geometry of the layers is used to emphasise the synthetic nature of the material where the colour or colours, spatially or temporally separated, alone may not be sufficient.

Where a single marking layer is used, this may occupy a substantial volume of the whole layer, object or synthetic gemstone. Where multiple layers are used in one or more groups, then the lower bound of the thickness of the layers would typically exceed $0.1\text{ }\mu\text{m}$, more typically exceed $1\text{ }\mu\text{m}$, even more typically exceed $2\text{ }\mu\text{m}$, and most typically exceed $5\text{ }\mu\text{m}$, and the upper bound of the thickness of the layers would typically be less than $100\text{ }\mu\text{m}$, more typically be less than $50\text{ }\mu\text{m}$, even more typically be less than $20\text{ }\mu\text{m}$ and most typically be less than $10\text{ }\mu\text{m}$, the basic criteria

being layers that are thin enough so as not to significantly colour the cut stone when viewed under normal light, whilst being sufficiently thick to provide sufficient visibility under the selected excitation wavelengths. Strictly a more fundamental quantity is dependent on the concentration of the defect centers responsible for the luminescence or phosphorescence in the marked layers.

In particular, viewing near normal to the plane of the marked layers using sub band-gap radiation, the critical parameter would be the concentration of irradiating centres (which would generally be proportional to the concentration of the relevant impurities) through the thickness of the layer projected onto the plane of the layer. That is, the observed brightness of the layer would be proportional to the product of the intensity of the exciting radiation, the concentration per unit volume of the irradiating centre, and the thickness of the layer. Other effects can also contribute to the observed intensity, including the effect of self absorption elsewhere in the material. Thus thicker layers would be preferred where concentrations of dopants were very low.

In contrast, viewing the edges of the layers in above band-gap excitation, the depth of material sampled would be largely fixed by the radiation wavelength and thus the observed brightness would be proportional to the product of the intensity of the exciting radiation and the concentration per unit volume of the relevant defect centers, with the thickness of the layer determining the lateral extent of the area emitting. Thicker layers may again assist in improving visibility where concentrations of dopants are low, by increasing the area to be observed.

Suitable sub band-gap wavelengths for viewing the marks can be determined as follows. Orange luminescence can be excited by a range of wavelengths such as 514 nm, 488 nm and shorter wavelengths, but excitation efficiency is reduced as wavelengths approach the UV. In contrast, blue-band luminescence is more efficiently excited by wavelengths approaching the long UV, such as the mercury lines at 369 nm

and 254 nm. These trends in the efficiency of excitation are not particularly limiting, however, and a range of wavelengths can be used to excite both emissions sufficiently well.

Typical above band-gap irradiation for viewing would be 193 nm. Generally high luminescence sources can be used and good spatial resolution still obtained because of the strong attenuation in the diamond. An instrument particularly suited to precise viewing and characterisation of the layers using above bandgap UV radiation is the 'Diamond View'TM instrument, developed by the Diamond Trading Company which combines a suitable UV source with digital image capture and allows the study of both fluorescence and phosphorescence in diamond samples even down to relatively low luminosity levels.

A particularly advantageous form of inexpensive viewer for general detection of the presence of the mark in this invention would combine a small box to fit over the CVD diamond layer, object or synthetic gemstone to exclude ambient light, with an excitation light source entering the box and a viewing window, possibly in the form of a magnifying lens, with a filter to remove the excitation wavelength. Alternatively ambient white light could also be removed by filters rather than excluded from the viewing box, with the filters then being essentially band-pass filters for the orange luminescence and blue-band phosphorescence.

The characteristic pattern of lines needs to be as widely applicable as possible, and potentially could be used in CVD diamond layers which are themselves boron doped to provide a visible blue colour, but also providing a source of blue-band phosphorescence, or in CVD diamond layers or objects which otherwise contain nitrogen and thus show orange luminescence. By combining the two layers together into a pattern, the same pattern can then be observed and identified in both these types of CVD diamond layer or object, with the risk that final layers at the edge of the mark may blend into the background. This risk is minimised by using an asymmetric mark, where one edge of the mark is defined by blue

phosphorescence and the other is defined by orange luminescence. Alternatively some neutral background could be used around the mark.

In CVD diamond layers with a sufficiently high level of boron more generally present through the volume, for example where a strongly coloured stone is required, then the addition of nitrogen in marker layers may not be sufficient to generate orange luminescence but may merely modulate the blue phosphorescence from donor-acceptor pair recombination. One solution is to specifically reduce the B concentration in the N doped layers to enable the orange luminescence to be observed. Alternatively, under such circumstances the modulation of the blue phosphorescence may be sufficient, and can be controlled by both the added N and added B concentrations.

There are further advantages to the choice of marker layers, in that the blue phosphorescence of the boron is stable with respect to post treatments such as annealing, so that these marks would remain even if the CVD diamond layer, object or synthetic gemstone was treated by such means. Conversely the orange luminescence is modified by annealing, particularly at very high temperatures. These lines would therefore indicate that the object had been post treated after the point of sale. In particular, annealing of the orange luminescence can convert the orange luminescence to a characteristic green luminescence or phosphorescence (the extent to which this light continues to be emitted after the removal of the excitation source varying over several orders of magnitude depending on the relative concentration of the defects involved). The stability of the layers showing blue phosphorescence thus enables the location of the previously orange luminescing bands to be determined and the treatment conditions to be determined from the modified, increased or reduced colour then present in these bands.

The simplest pattern of multiple lines envisaged using one type of marker layer (e.g blue phosphorescence) is shown in Figure 1. The diamond

material 10 includes a pair of marker layers 12,14 separated by a spacer layer 16.

Here, t_m is the thickness of the respective marker layers 12,14 and t_s is the thickness of the spacer layer 16.

Adding in a second type of layer (e.g. orange luminescence) gives a structure as shown in Figure 2. In this embodiment, the synthetic diamond material 20 has a pair of first marker layers 22,24 separated by a second marker layer 26.

In particularly preferred embodiments, it is envisaged that the structures would probably be more complex, using more layers and with clearly varying thicknesses. In the synthetic diamond material 30 shown in Figure 3, the thicknesses of the layers 32, 34, 36, 38, 40 and 42 are varied. They may be, for example, 50 μm (34,40), 25 μm (36,38), and 12 μm (32,42), giving a total thickness of the mark of 175 μm , which would be clearly visible under the correct viewing illumination provided that the dopant levels were suitably controlled.

It is believed that the marker layers of given thicknesses can be grown to an accuracy of 10% or better. For thicker layers or in a routine production process it may be possible to achieve an accuracy of 5% or better. However, when viewing the layers with above band-gap illumination, the marker layers will be seen on any surface which intersects those layers, but that surface may not be normal (i.e. at right angles) to the marker layers, and in many commercial diamond objects including synthetic gemstones this will often be the case. Thus, the absolute dimensions of the layers will typically not be consistent from one facet of a CVD diamond object to another, or necessarily between similar objects (although this may be the case if both the marker layers and the orientation of the object cut out of the diamond are crystallographically oriented), easily varying by up to about +/- 50% depending on the angle of the facet on which they are viewed. What will be consistent across any single facet, however, is the relative ratios of

the thicknesses of the layers and the sequence of colours, which will allow for proper identification of the mark of origin or fingerprint. It is of course possible from the geometry of the CVD diamond object and the specific orientations of the layer(s) and the facet intersected, to calculate the precise thicknesses of the layers, but this is a level of complexity which the preferred embodiment avoids.

Thus, taking relative ratios of the thicknesses of layers as the only measurable characteristic, a single marker layer gives no information, since there is no reference point. However, a mark of origin with 2 marker layers and a spacer layer gives two unique parameters, for example taking the spacer layer as the scale bar against which to compare the thickness of each of the marker layers. A mark of origin with 3 marker layers and 2 spacer layers gives 4 unique parameters (provided there is no mirror symmetry), etc. Hence, in practice, it is believed that the reasonable number of layers to provide a distinguishable mark of origin but permitting several deliberate variants would be three marker layers, giving 4 unique thickness ratio parameters. In the case where 2 distinct type of marker layers are used alternately, the number of unique parameters can be considered in a similar fashion.



Fig.1

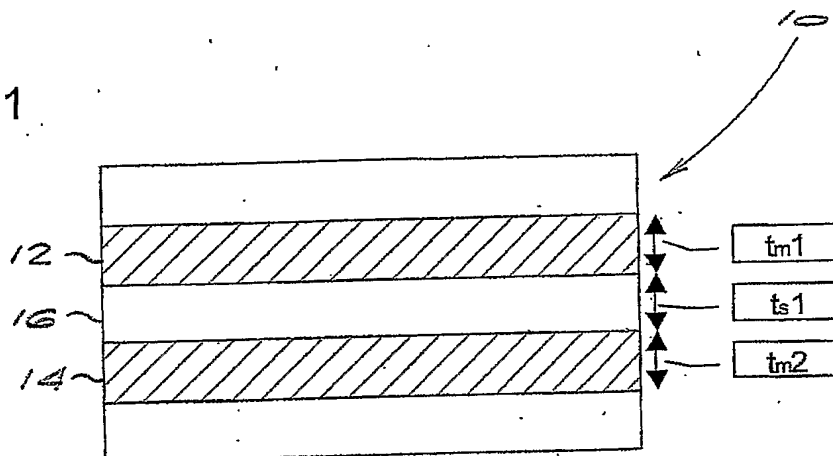


Fig.2

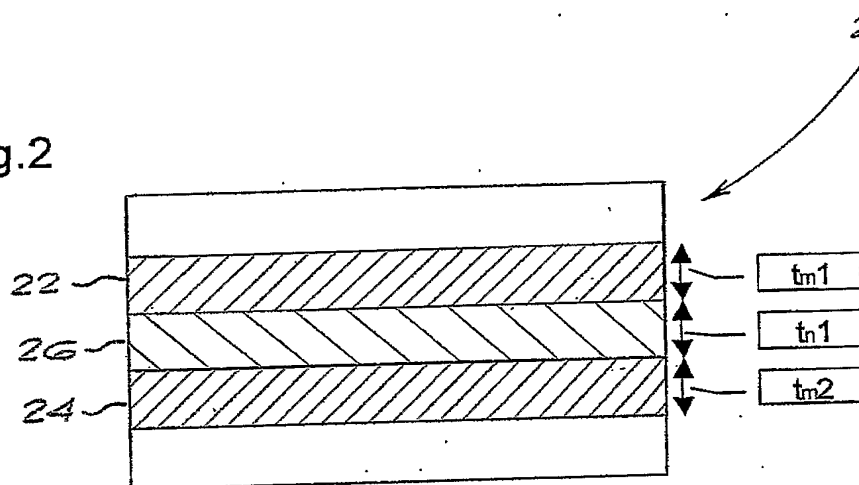
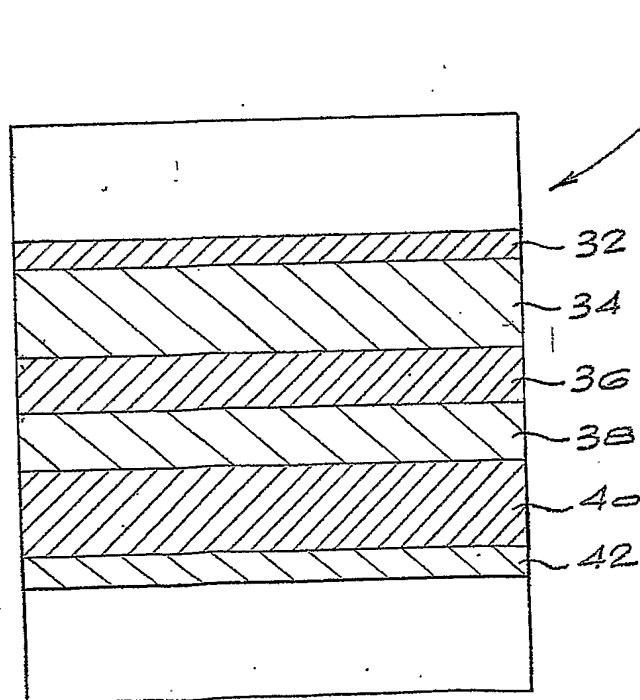


Fig.3



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